

HIGH PRODUCTION VOLUME (HPV) CHEMICAL CHALLENGE PROGRAM

TEST PLAN

LUBRICATING OIL BASESTOCKS CATEGORY

Submitted to the US EPA

by

The Petroleum HPV Testing Group

www.petroleumhpv.org

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Plain Language Summary

This test plan addresses the petroleum refinery streams known as lubricating base oils, also referred to as base oils or lubricating basestocks. Base oils are the primary hydrocarbon components of industrial lubricants including engine oils, transmission fluids, hydraulic fluids, gear oils, metalworking oils, greases, heat transfer oils, general-purpose oils, and machine oils. The more intensively refined base oils (reduced levels of undesirable components) are used as food machinery lubricants, pharmaceutical white oils, laxatives, body lotions, cosmetics, direct food additives, and in a number of food-contact applications.

The materials in this category are complex petroleum mixtures composed primarily of saturated hydrocarbons with carbon numbers ranging from C15 to C50. At ambient temperatures lubricating base oils are liquids of varying viscosities, with negligible vapor pressures. Base oils are produced by first distilling crude oil at atmospheric pressure to remove lighter components (e.g. gasoline and distillate fuel components), leaving a residue (residuum) that contains base oil precursors. This atmospheric residuum is then distilled under vacuum to yield a range of distillate fractions (unrefined distillate base oils) and a vacuum residuum. Removal of the asphalt components of the vacuum residuum results in unrefined residual base oils. These distillate and residual base oil fractions may then undergo a series of extractive or transforming processes that improve the base oils' performance characteristics and reduce or eliminate undesirable components.

Given their process histories, compositional differences, and physicochemical differences, the streams within this HPV category can be divided into two subcategories:

- Distillate base oils, and
- Residual base oils.

The distillate base oils can be further grouped by degree of processing, levels of unwanted constituents, and expected mutagenicity and carcinogenicity potential:

- Unrefined & mildly refined distillate base oils, and
- Highly & severely refined distillate base oils.

Unrefined & mildly refined distillate base oils contain the highest levels of undesirable components, have the largest variation of hydrocarbon molecules and have shown the highest potential carcinogenic and mutagenic activities. Highly & severely refined distillate base oils are produced from unrefined & mildly refined oils by removing or transforming undesirable components. In comparison to unrefined & mildly refined base oils, the highly & severely refined distillate base oils have a smaller range of hydrocarbon molecules and have demonstrated very low mammalian toxicity. Mutagenicity and carcinogenicity testing of residual oils has been negative, supporting the Testing Group's belief that these materials lack biologically active components or the components are largely non-bioavailable due to their molecular size.

The Testing Group is proposing to perform a reproductive/developmental screening study (OECD 421) on a representative sample of a highly to severely refined distillate base oil (other than a food/drug grade white mineral oil). The limited reproductive and developmental data for highly & severely refined distillate base oils, coupled with positive effects for this endpoint in a study of heavy vacuum gas oil (a material similar to an unrefined distillate base oil) suggest the need for additional data on the highly & severely refined distillate oils.

The Testing Group is also proposing to perform a repeat-dose/reproductive/developmental screening study (OECD 422) on a representative sample of the residual base oils. The lack of adequate documentation of a complete data set on the repeat-dose endpoints, and the lack of reproductive and developmental toxicity endpoints for residual base oils suggest the need for a screening study on this subcategory of materials.

The Testing Group believes conducting these two studies will allow the Group to:

- Complete the SIDS (Screening Information Data Set) characterization of the mammalian toxicity of materials within the lubricating oil basestocks category, and
- Test the Group's hypotheses that:

- The reproductive and developmental toxicity of the distillate base oils is inversely related to the degree of processing, and
- The residual base oils lack repeat-dose, reproductive and developmental toxicity potential.

When physicochemical data did not exist or was impractical to obtain for the lubricating oil basestocks category, calculated physicochemical and environmental data for selected constituents of lubricating oil basestocks has been developed using the EPIWIN© computer model.

Substantial data was reviewed on the eco-toxicological characteristics of distillate and residual base oils. For fish, invertebrates and algae, no acute toxicity was measured in any of 20 tests. Furthermore, no chronic toxicity to aquatic invertebrates was found in 10 of 11 studies examined. The Testing Group thinks the existing ecotoxicity data on lubricating oil basestocks adequately describes their potential toxicity. Therefore the Testing Group is not proposing any additional ecotoxicity testing on any of the lubricating oil basestocks category members.

Description of the Lubricating Oil Basestocks Category

The Lubricating Oil Basestocks category includes both refinery streams and finished products. The materials in this category are complex petroleum mixtures that boil between 700 and 1000°F (371-538°C) and are composed primarily of saturated hydrocarbons with carbon numbers ranging from C15 to C50. The molecular makeup of these oils consists of paraffinic, isoparaffinic, naphthenic, and aromatic hydrocarbon groupings that are varied in complexity and number. At ambient temperatures, all the materials in the category are liquids of varying viscosities with negligible vapor pressures and water solubility values. Because they are complex mixtures, the lubricating oil basestocks are typically not defined by detailed compositional information but instead by process history, physical properties, and product use specifications. Whereas detailed compositional information may be limited, general compositional information can be inferred from the base oil's physical properties – e.g. the higher the boiling temperature range of a fraction, the higher the molecular weight of the oil's components.

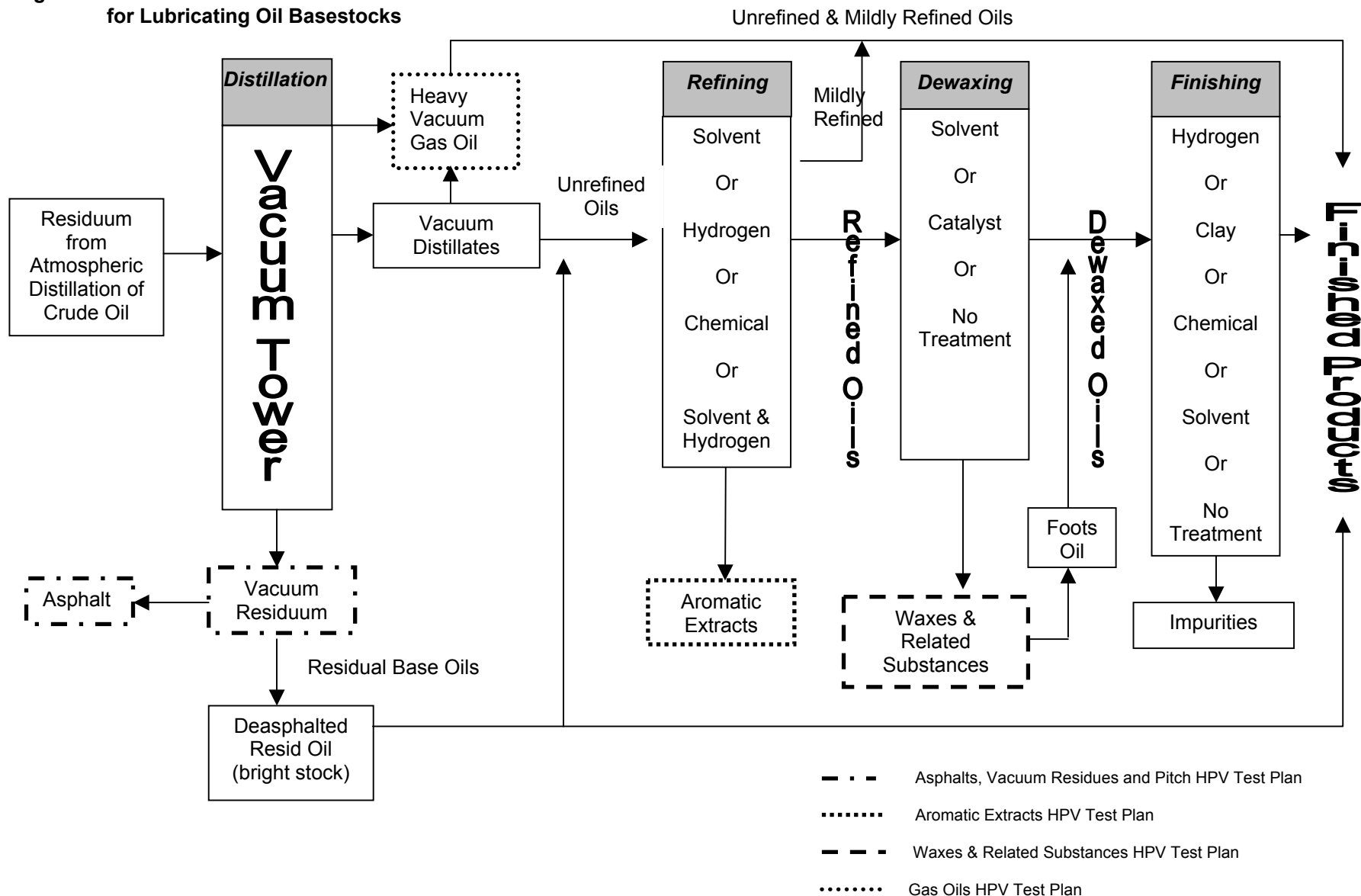
As shown in Figure 1, the materials included in the category are produced by the vacuum distillation of the residuum that results from the atmospheric distillation of crude oil. This vacuum distillation produces a range of distillate fractions (unrefined distillate base oils) and a vacuum residuum. As the boiling ranges of the fractions increase, the levels of polycyclic aromatic compounds (PACs), polycycloparaffins and heteratoms (N, O, S, and metals) increase, while the levels of paraffins decrease. This is shown schematically in Figure 2.

Removal of the asphalt components (e.g., asphaltenes, resins) of the vacuum residuum results in unrefined residual base oils. The unrefined distillate and residual base oils can either be blended into other process streams, or undergo additional refining in order to produce “finished” base oils. The additional refining consists of a series of extractive or transforming processes that improve the base oils' performance characteristics and remove, reduce or transform undesirable components. An example of the transformations that take place is that of hydrocracking, a process in which aromatics are converted to naphthenics and paraffins by catalytically breaking carbon-carbon bonds under high-pressure hydrogen.

The removed and transformed materials are deemed undesirable because they are either deleterious to product performance and/or are potentially carcinogenic. The undesirable components include aromatics, metals, waxes, and trace components causing unwanted colors or odors (i.e. sulfur). The aromatics include polycyclic aromatic compounds [PACs], some of which are heterocyclics, PACs with inclusions of N, S, O. Premium lube base stocks have low levels of nitrogen and sulfur, the hydrocarbons present being predominantly naphthenics and isoparaffins. Naphthenics have good low temperature viscosity and oxidative stability while isoparaffins possess excellent oxidation stability, good viscosity characteristics, and low volatility. Normal paraffins, however, have poor low temperature properties. Aromatics have high volatility and poor oxidative stability in most lubricant applications, and therefore, processing is directed toward reducing the aromatics content. Sulfur causes deposits, off color, and odor, while nitrogen causes deposits and promotes oxidation.

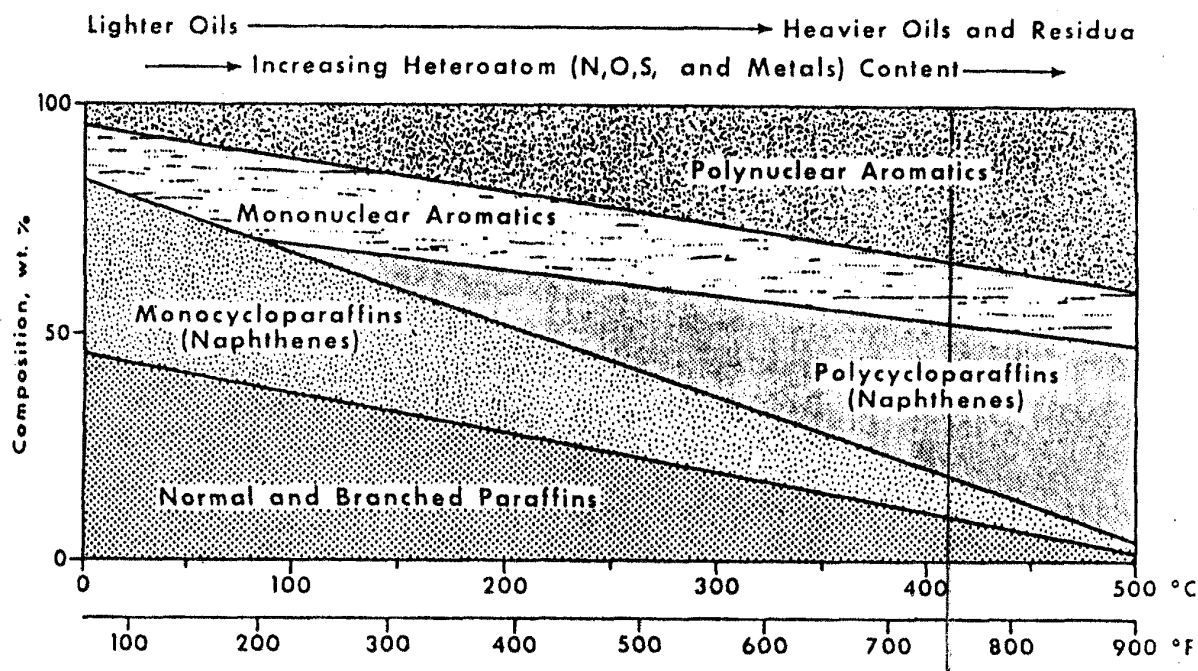
The more extensive the extractive and transforming processes an oil undergoes, the more “severe” is the oil's processing. Terms such as “mildly” and “highly” are also used to describe the degree of processing. Within the base oil category, the unrefined base oils contain the highest levels of undesirable components, have the largest variation of hydrocarbon molecules and in the case of distillate base oils, have shown the highest potential carcinogenic and mutagenic activity. Because of the “subtractive” nature of base oil processing, streams that have been “severely” or “highly” processed have much lower levels of undesirable components, a narrower range of hydrocarbon molecules and have demonstrated very low toxicity.

Figure 1. Production Process Schematic for Lubricating Oil Basestocks



Links to resources containing additional details regarding lubricating oil basestocks refining and petroleum processes in general can be found in Appendix B “Links to Additional Resources”.

Figure 2. Refinery Stream Composition – Boiling Range vs. General Composition



Mobil, 1997

Based on the vacuum tower distillation fraction from which they originate and their corresponding physical-chemical properties, the thirty-six substances (see Appendix 1) included in the Lubricating Oil Basestocks Test Plan can be divided into two subcategories:

- Distillate base oils, and
- Residual base oils.

The distillate base oils can be further divided by degree of processing, levels of unwanted constituents, and expected mutagenicity and carcinogenicity potential into:

- Unrefined & mildly refined, and
- Highly & severely refined base oils.

Physical-chemical properties for selected base oils can be found in Table 1.

Table 1. Physical-chemical Properties of Selected Lubricating Oil Basestocks

| Base oil description | Kinematic viscosity * | | Flash Point (°C) | Pour Point (°C) | Density (kg/l) | Average Molecular Weight |
|--|------------------------------|-------------------------------|------------------|-----------------|----------------|--------------------------|
| | at 40°C (mm ² /s) | at 100°C (mm ² /s) | | | | |
| Distillate oils | | | | | | |
| Solvent-dewaxed, light paraffinic (64742-56-9) | 8.4 | 2.4 | 157 | -18 | 0.85 | 280 |
| Solvent-dewaxed, heavy paraffinic (64742-65-0) | 25.1 | 4.8 | 204 | -12 | 0.86 | 390 |
| Hydrotreated, light paraffinic (64742-55-8) | 17.0 | 3.7 | 190 | -18 | 0.86 | 360 |
| Hydrotreated, heavy paraffinic (64742-54-7) | 73.9 | 9.1 | 232 | -9 | 0.88 | 500 |
| Hydrotreated, light naphthenic (64742-53-6) | 8.5 | 2.2 | 145 | -60 | 0.87 | 290 |
| Hydrotreated, heavy naphthenic (64742-52-5) | 145 | 10.5 | 220 | -24 | 0.91 | 440 |
| White mineral oil (8042-47-5) | 27.3 | 5.0 | 217 | -15 | 0.86 | 400 |
| | | | | | | |
| Residual oils | | | | | | |
| Solvent-dewaxed (64742-62-7) | 1300 | 50 | 285 | -6 | 0.95 | 700 |

*Kinematic viscosity is often expressed in Centistokes (cSt), 1 mm²/second (mm²/s) = 1 cSt.

CONCAWE, 1997

Distillate Base Oils

Distillate base oils contain components whose boiling points typically range from 300 to 600°C (CONCAWE, 1997). These materials are found in the majority of the lubricating products sold to the public. Distillate base oils are often described as either "naphthenic" (saturated ring hydrocarbons) or "paraffinic" (straight or branched chain hydrocarbons) depending on their crude source and/or the dominant hydrocarbons present. The difference between naphthenic and paraffinic base oils is one of relative percentage since, naphthenes and paraffins are present in both types of oils. Thus, a base oil might be called a paraffinic oil if it is 60% paraffins and 30% naphthenes or a naphthenic oil if it is 60% naphthenes and 30% paraffins. Base oils are also often described as either "light" (viscosity less than 19 cst @40°C) or "heavy" (viscosity greater than 19 cst@40°C). The naphthenic/paraffinic and light/heavy nomenclatures are primarily used to distinguish product application and lubricant quality parameters rather than health and safety characteristics, since a significant amount of toxicology data exists that shows little differentiation between these four classifications (see Appendix C, Robust Summary).

For the purposes of the HPV program, a more critical defining feature of the distillate base oils is the severity of the processing an oil has undergone. The focus on the degree of processing is supported by the physical-chemical characteristics of the materials, the subtractive nature of the processing the base oils undergo, and the existing toxicology database. Numerous tests have shown that a lubricating base oil's mutagenic and carcinogenic potential correlates with its 3-7 ring PAC content, and the level of DMSO

extractables, both characteristics that are inversely related to the degree/conditions of processing (Doak, et al., 1983; Halder, et al., 1984; IARC, 1984; Kane, et al., 1984; Singer, 1986; Chasey, et al., 1993; Roy, et al. 1988, 1996; Blackburn, et al., 1984; 1986; 1996; CONCAWE, 1994; EU, 1994). Based on the results published by Feuston, et al. (1994), the Testing Group thinks the same inverse relationship may exist for subchronic and other non-carcinogenic endpoints.

Using the extent of refining as a criterion, materials in the distillate base oils sub-category can be divided into two groups:

- Unrefined & mildly refined distillate base oils, and
- Highly & severely refined distillate base oils.

The unrefined & mildly refined distillate oils receive no or minimal treatment beyond the initial vacuum distillation. Consequently, they contain the highest levels of undesirable components, have the largest variation of hydrocarbon molecules and have shown the highest carcinogenic and mutagenic activity. Unrefined & mildly refined distillate oils are primarily used as feedstocks for the production of more highly refined base oils. They may also be used in a small number of applications such as certain general-purpose lubricants that are consumed in use (once-through lubricants) and heat treatment oils. Because human exposures to unrefined and mildly refined distillate base oils occur in occupational settings, human exposures are restricted to droplet aerosols, liquid on the skin and occasional accidental ingestion. However, these oils are all clearly labeled by the manufacturers as potential carcinogens and hazardous to health.

Refined distillate base oils are produced from unrefined base oil fractions by undergoing additional processing designed to reduce or transform the undesirable components (see Figure 1). In general, each additional step of processing (increasingly severe processing) results in:

- Lower levels of unwanted components; including aromatics, metals, waxes, and trace components causing unwanted colors or odors (i.e. sulfur), -
- A narrower range of hydrocarbon molecules (increasing concentration of paraffins and naphthenes), and
- Lower, if any, carcinogenic or mutagenic activity.

Some distillate base oils are destined for use in food, food contact, cosmetic, pharmaceutical and related applications. Known as white oils, these very severely refined distillate base oils undergo numerous processing steps that essentially eliminate or transform all undesired components, including unsaturated hydrocarbons and aromatics. When used in food, food contact, cosmetic, pharmaceutical and related applications, base oils have to meet stringent purity requirements as described in the respective national Pharmacopoeia and international legislations. These regulations generally specify melting ranges, color, polycyclic aromatic hydrocarbon content and other impurity limits (U.S. FDA, 2002; USP, 2002; CONCAWE, 1984, JECFA 2002).

Almost all commercial base oils used in the United States are highly refined. Others could be considered severely refined, yet are not medicinal, cosmetic or food grade white oils. Refined lubricants destined for commercial or industrial applications (non-medicinal/non-food/non-cosmetic) are not processed to the same level of severity as the white oils – thus leaving these highly refined base oils with very low, but measurable levels of sulfur and aromatics. These highly refined base oils display toxicological properties much closer to the food/drug/cosmetic grade white oils than they do to the unrefined & mildly refined base oils.

Detailed compositional and physical-chemical properties for two representative samples of materials that represent the boundaries of the distillate base oils sub-category are shown in Table 2.

Table 2. Physical-chemical Properties of Representative Distillate Base Oil Samples

| | Unrefined | Severely Refined (medicinal grade) |
|--|------------------|--|
| Avg Molecular Weight (gm/mol) | 300 | 320 |
| Density @15°C | 0.8651 | 0.857 |
| Viscosity @40°C (centistokes) | 14.07 | 13.3 |
| Viscosity @100°C (centistokes) | 2.79 | 3.08 |
| Pour Point (°F) | + 60 | -32.8 |
| Distillation °F @ 760mm | | |
| 5% (vol) | 658 | 509 |
| 50% | 711 | 689 |
| 95% | 790 | 833 |
| Refractive Index RI units @20°C | 1.4815 | 1.4688 |
| Total Sulfur (wt %) | 0.38 | <.0001 |
| Heavy Metals Total mg/kg | <1 | <1 |
| Hydrocarbon Type | | |
| Nonaromatics (wt %) | 79.1 | 100 |
| Aromatics (wt %) | 20.9 | <2x10 ⁻⁵ |

API, 1987b
CONCAWE, 1993

Residual Base Oils

Residual base oils are derived from the residuum of the vacuum distillation tower and may contain components boiling as high as 800°C (CONCAWE, 1997). As can be seen from Table 1, the residual oils have molecular weights that are much higher than the distillate base oils. Residual base oils are primarily used in situations requiring oils with a high viscosity, e.g., gear oils.

As shown in Figure 2, residual oils have substantial PAC levels when assayed by traditional methods. On this basis, they would be expected to have mutagenic and/or carcinogenic activity. However, no adverse effects have been seen in either *in vitro* mutagenicity or dermal carcinogenicity testing of residual base oils, irrespective of the degree of processing they have undergone. Ultraviolet, HPLC/UV, GC/MS, and infrared analyses of these oils indicate that the aromatics they contain are predominantly 1-3 rings that are highly alkylated (paraffinic and naphthenic). Because they are found in such a high boiling material (> 1070°F), it is estimated that the alkyl side-chains of these 1-3 ring aromatics would be approximately 13 to 25 carbons in length. These highly alkylated aromatic ring materials are either devoid of the biological activity necessary to cause mutagenesis and carcinogenesis, or are largely non-bioavailable to the organisms (Roy, et al., 1988).

Category Rationale and Test Material Description

The Testing Group made the following assumptions when analyzing the existing data, proposing testing and selecting test materials:

- The materials included in the Lubricating Base Oils category are related from both process and physical-chemical perspectives;
- The potential toxicity of a specific distillate base oil is inversely related to the severity or extent of processing the oil has undergone, since:

- The adverse effects of these materials are associated with undesirable components, and
- The levels of the undesirable components are inversely related to the degree of processing;
- Distillate base oils receiving the same degree or extent of processing will have similar toxicities;
- The potential toxicity of residual base oils is independent of the degree of processing the oil receives.

The Testing Group is proposing to perform a reproductive/developmental screening study (OECD 421) on a representative sample of a highly to severely refined distillate base oil (other than a food/drug grade white mineral oil). The limited reproductive and developmental data for highly & severely refined distillate base oils, coupled with positive effects for this endpoint in a study of heavy vacuum gas oil (a material similar to an unrefined distillate base oil) suggest the need for additional data on the highly & severely refined distillate oils.

The Testing Group is also proposing to perform a repeat-dose/reproductive/developmental screening study (OECD 422) on a representative sample of the residual base oils. The lack of a complete data set on the repeat-dose, reproductive and developmental toxicity endpoints for residual base oils suggest the need for a screening study on this subcategory of materials.

The Testing Group is not proposing any toxicity testing of unrefined & mildly refined oils. While no reproductive or developmental toxicity tests have been performed on these materials, the Testing Group believes the reproductive and developmental effects of these oils will be similar to those shown by heavy vacuum gas oil, a material similar to an unrefined distillate base oil. In comparison to the other members of the category, both heavy vacuum gas oil and the unrefined & mildly refined oils have higher levels of biologically active and available components.

The Testing Group believes conducting these two studies will allow the Group to:

- Complete its characterization of the mammalian toxicity endpoints in the Screening Information Data Set (SIDS) for the of materials within the lubricating oil basestocks category, and
- Test the Group's hypotheses that:
 - The distillate base oils reproductive and developmental toxicity potential is inversely related to the degree of processing, and
 - The residual base oils lack repeat-dose, reproductive and developmental toxicity potential.

Specific analytical data on the two base oil test samples will be available when the samples are obtained. The distillate oil test sample will have physicochemical and compositional properties similar to those shown in Table 2. For the residual oil test sample, the Testing Group will attempt to select an oil that has received a minimal amount of processing.

No additional mammalian or environmental fate/effects testing is proposed for the lubricant base oil category.

Evaluation of Existing Health Effects Data and Proposed Testing

General Evaluation

Many studies have been reported for this category of materials, ranging from acute to long-term carcinogenicity studies. Additional reviews by various individual authors and expert panels have also been published (Bingham et al., 1980; WHO, 1982; IARC, 1984; API, 1992; SCF, 1995; JECFA, 1996; CONCAWE, 1997). The toxicity testing has consistently shown that lubricating base oils have low acute toxicities. Numerous tests have shown that a lubricating base oil's mutagenic and carcinogenic potential correlates with its 3-7 ring PAC content, and the level of DMSO extractables (e.g. IP346 assay), both

characteristics that are directly related to the degree/conditions of processing (Halder, et al., 1984; IARC, 1984; Kane, et al., 1984; Chasey, et al., 1993; Roy, et al. 1988, 1996; Blackburn, et al., 1984, 1986; 1996; CONCAWE, 1994; EU 1994).

The Test Plan addresses the health effects endpoints of the category by:

- Evaluating the extensive toxicology database for the lubricating base oils,
- Using read-across information whenever possible among category members, and
- Proposing the minimal amount of mammalian toxicity testing needed to characterize the category and test the Testing Group's hypotheses:
 - The distillate base oils reproductive and developmental toxicity is inversely related to the degree of processing, and
 - The residual base oils lack reproductive and developmental toxicity potential.

Acute Toxicity

Distillate Base Oils

Unrefined & Mildly Refined

LD₅₀s of >5000 mg/kg (bw) and >2g/kg (bw) for the oral and dermal routes of exposure, respectively, have been observed in rats dosed with an unrefined light paraffinic distillate (API, 1986d). The same material was also reported to be "moderately irritating" to the skin of rabbits (API, 1986d). When tested for eye irritation in rabbits, the material produced Draize scores of 3.0 and 4.0 (unwashed/washed eyes) at 24 hours, with the scores returning to zero by 48 hours (API, 1986d). The material was reported to be "not sensitizing" when tested in guinea pigs (API, 1986d).

Highly & Severely Refined

Multiple studies of the acute toxicity of highly & severely refined base oils have been reported. Irrespective of the crude source or the method or extent of processing, the oral LD₅₀s have been observed to be >5 g/kg (bw) and the dermal LD₅₀s have ranged from >2 to >5g/kg (bw) (API, 1986c; CONCAWE, 1997). The LC₅₀ for inhalation toxicity ranged from 2.18 mg/l to >4 mg/l (API, 1987a; CONCAWE, 1997). When tested for skin and eye irritation, the materials have been reported as "non-irritating" to "moderately irritating" (API, 1986c; CONCAWE, 1997). Testing in guinea pigs for sensitization has been negative (API, 1986c; CONCAWE, 1997).

Residual Base Oils

There are no acute toxicity data available for the residual base oils. The Testing Group thinks the high molecular weight of these materials and associated low bioavailability preclude the systemic doses necessary to produce acute toxicity. Furthermore, tests of a variety of distillate base oils, including unrefined materials that contain high levels of biologically active materials, have consistently shown low acute toxicity.

Summary: No additional testing is planned. The Testing Group thinks the existing data is sufficient to characterize the acute toxicities of this category of materials. Multiple acute toxicity studies have been reported on a variety of distillate base oils. The studies have consistently found these materials to have low acute toxicities. The Testing Group believes there is no need for acute toxicity testing of residual oils given their low potential for human exposure, reported low bioavailability and lack of genotoxicity and dermal carcinogenicity.

Repeat-Dose Toxicity

Distillate Base Oils

Unrefined & Mildly Refined

Two hundred, 1000 and 2000 mg/kg (bw)/day of an unrefined base oil has been applied undiluted to the skin of male and female rabbits (API, 1986b). The test material was applied to the rabbits' skins 3 times/week for 4 weeks. To ensure maximum exposure, the applied material was covered with an occlusive dressing for 6 hours. In the high dose group, body weight gains were affected by treatment. These effects were largely due to effects on growth rate during the first week of the study. There were no significant differences between treated and control groups for any of the recorded hematological and clinical chemistry values. Gross and microscopic pathology findings relating to the treated skin were seen in all rabbits in the highest dose group. The findings consisted of "slight" to "moderate" proliferative changes in the treated skin.

Highly & Severely Refined

Two hundred, 1000 and 2000 mg/kg (bw)/day of a highly to severely refined base oil has been applied undiluted to the skin of male and female rabbits (API, 1986a). The test material was applied to the rabbits' skin 3 times/week for 4 weeks. To ensure maximum exposure, the applied material was covered with an occlusive dressing for 6 hours. Minimal to moderate skin irritation was observed in the various base oil dose groups. Throughout the study, male and female body weights in the high dose group and female body weights in the mid dose group were reduced when compared to their respective controls. Body weight gains observed in both male and female high dose groups were significantly lower than control values. At necropsy, the absolute testis weights and the relative weights of the right testis were lower in the high dose males than in controls. A common finding at necropsy in all the treatment groups was dry, scaly, rough, fissured, crusted and/or thickened skin. Histopathological examination revealed "slight" to "moderate" proliferative changes in the skin in all rabbits in the high dose group. These changes were accompanied by an increased granulopoiesis of the bone marrow. The testes of the majority of males in the high dose group had bilateral diffuse tubular hypoplasia accompanied by aspermatogenesis and atrophy of the accessory sex organs.

Repeat-dose dermal studies in rabbits have been reported for 15 different highly to severely refined base oils (all 28 day except one 21 day study) (CONCAWE, 1997). Doses have ranged from 200 to 5000 mg/kg/day, 3 times weekly. The primary effect in these studies was skin irritation, which ranged from "non" to "moderate". Systemic effects were observed in only one study. The effects consisted of a single incident of decreased body weight and increased relative liver weight, elevated SGOT and SGPT values and a sub-acute hepatitis.

Three highly to severely refined base oils have been tested in a four-week inhalation study (Dalbey, et al., 1991). Groups of male and female Sprague-Dawley rats were exposed to a base oil aerosol at nominal concentrations of 0, 50, 220 and 1000 mg/m³. Exposures were for 6 hours/day, 5 days/week. Apart from occasional diarrhea, there were no treatment related clinical observations and body weights were unaffected by exposure. No treatment related effects were found in any of the hematological or clinical chemical parameters that were measured. There was also no treatment related effect on sperm morphology. Wet and dry lung weights were increased in a dose related manner. For both males and females, the ratios of wet to dry lung weights were significantly increased at the highest dose concentrations of all three base oils. Treatment related changes were observed microscopically in the lungs and tracheobronchial lymph nodes of many of the treated animals. These changes consisted of the presence in the alveolar spaces of foamy macrophages with numerous vacuoles of varying size.

Subchronic oral toxicity studies have been carried out on six different food grade white oils (BIBRA, 1992). For ninety days, male and female Fischer 344 rats were fed diets containing severely refined base oils (white oils) at concentrations of 0.002, 0.02, 0.2 and 2.0% (w/w). The effects observed in the study were inversely related to the molecular weights of the six oils. The highest molecular weight material produced no effects other than increases (approximately 10%) in both food consumption and ASAT values in the males of the highest dose group. Histological examination revealed a small amount of mineral hydrocarbon in the livers of the male rats in the highest dose group. The lowest molecular weight base oil produced a similar increase in food consumption amongst males in the highest dose group. Treatment related increases in organ

weights (absolute and relative) and selected hematology and clinical chemistry values were also seen in both males and females. Histological examination of tissues from the high dose female group found a significant increase in hepatic granulomas and vacuolation of the lamina propria of the ileum and jejunum. In females, a treatment related histiocytosis was observed in the mesenteric lymph nodes beginning at the 0.02% dose level. Treatment related histiocytosis was also observed in the mesenteric lymph nodes of males, but was not observed below the 0.2% dose level. The study authors concluded that the LOEL values for five of the six base oils ranged from 0.002% to 0.02% dietary base oil concentrations. The NOEL for the oil with the highest molecular weight was a 2.0% dietary concentration.

In addition to the report summarized in the preceding paragraph, numerous repeat-dose oral toxicity studies on food-grade white oils have been reported in the open literature. Studies in Long Evans rats and Beagle dogs have reported no adverse effects (Bird, et al., 1990; McKee, et al., 1987b). Furthermore, studies with a low molecular weight white oil have demonstrated that the Fischer 344 rat is more sensitive in its response to mineral hydrocarbons than the Sprague Dawley rat (Firriolo et al., 1995).

The Testing Group believes the weight of evidence from all available data on highly & severely refined base oils support the Testing Group's presumption that a distillate base oil's toxicity is inversely related to the degree of processing it receives. The effects above do not contradict the Testing Group's hypothesis because:

- The granulomatous lesions induced by the oral administration of white oils are essentially foreign body responses. The lesions occur only in rats, of which the Fischer 344 strain is particularly sensitive,
- The testicular effects seen in rabbits after dermal administration of a highly to severely refined base oil were unique to a single study and may have been related to stress induced by skin irritation, and
- The accumulation of foamy macrophages in the alveolar spaces of rats exposed repeatedly via inhalation to high levels of highly to severely refined base oils is not unique to these oils, but would be seen after exposure to many water insoluble materials.

Residual Base Oils

No subchronic repeat-dose studies have been reported on residual base oils. However, two dermal carcinogenicity studies have been performed (see next section).

Carcinogenicity

Distillate Base Oils

Although carcinogenicity is not an official endpoint of the HPV program, the Testing Group notes that numerous carcinogenicity studies have been carried out on lubricating base oil samples, ranging from "unrefined" to "highly refined". Data from these studies have been reported and reviewed elsewhere (Bingham, et al., 1980; Blackburn, et al., 1984, 1996; CONCAWE, 1994; 1997; IARC, 1984; Roy, et al., 1988, EMBSI, 2001b; Shoda, et al., 1997). The general conclusions that can be drawn from the animal carcinogenicity studies are:

- Highly & severely refined base oils are not carcinogens, when given either orally or dermally.
- Unrefined & mildly refined base oils are potential skin carcinogens.
- When applied repeatedly to the skin, carcinogenic base oils are associated only with skin tumors and not with an increase in systemic tumors.

Residual Base Oils

A dermal carcinogenicity study of a residual base oil in mice has been reported by King (1991). The test substance was described as "a non-solvent refined, deasphalted, dewaxed residual paraffinic lubricant base oil". For eighteen months, three times/week, undiluted test material was applied to the

skin of female CF1 mice. Two other groups of mice underwent similar treatments, but for only 22 or 52 weeks. The base oil produced minimal clinical evidence of skin irritation. No tumors of epidermal origin were observed in animals dosed with the base oil. Furthermore, no treatment-related effects were observed with regard to clinical condition, body weight gain, mortality or post mortem findings. Due to the lack of experimental detail in the published report, the Testing Group is unable to assign a Klimisch reliability score to the study. The data from this study provide important information on potential carcinogenicity and selected repeat-dose parameters. However, the existing study documentation does not provide data for a number of the endpoints contained in the SIDS repeat-dose study designs.

A second dermal carcinogenicity study of a residual base oil has been conducted in male C3H/HeJ mice (Exxon, 1984). The test substance was described as "deasphalted, dewaxed, residual oil". The test material was applied undiluted to the animals' backs, three times/week for 24 months. None of the animals treated with the test material developed skin tumors, or any other tumors considered treatment-related. Due to the lack of experimental detail in the published report, the Testing Group is unable to assign a Klimisch reliability score to the study. The data from this study provide important information on potential carcinogenicity and selected repeat-dose parameters. However, the existing study documentation does not provide data for a number of the endpoints contained in the SIDS repeat-dose study designs.

The absence of systemic toxicity in these two dermal carcinogenicity studies supports the Testing Group's belief that the high molecular weight of the residual base oils and the resulting low bio-availability preclude the internal doses necessary to elicit systemic toxicity.

Summary: No additional testing is planned for distillate oils. The Testing Group proposes to test a representative sample of residual base oils by the dermal route of administration using a 28-day repeated-dose/reproductive/developmental toxicity screening protocol (OECD Test Guideline 422). Multiple repeat-dose toxicity studies, utilizing a variety of exposure routes, have been reported on a variety of distillate base oils. The Testing Group believes the existing data is sufficient to characterize the repeat-dose toxicity of the distillate base oils. The Testing Group is proposing a repeat dose test (OECD Test Guideline 422) for residual base oils because the documentation of existing carcinogenicity studies does not provide data for a number of the endpoints contained in the SIDS repeat-dose study designs.

Genotoxicity

***In-Vitro* (Mutagenicity)**

Distillate Base Oils

Unrefined & Mildly Refined

Modified Ames assays have been carried out on a number of base oils that were either unrefined or poorly refined. The oils were found to be mutagenic, with a strong correlation between mutagenicity and 3-7 ring PAC content (Blackburn, et al., 1986; Roy, et al., 1988).

Highly & Severely Refined

Several studies have reported the results of testing different base oils for mutagenicity using a modified Ames assay (Blackburn, et al., 1984, 1986; Roy, et al., 1988). Base oils with no or low concentrations of 3-7 ring PACs had low mutagenicity indices.

Residual Base Oils

Samples of a vacuum residuum and four residual base oils tested negative for the induction of frame shift mutations in modified Ames assays (EMBSI 2000, 2001a; Petrolabs, 1998; 2000). The Testing Group is unable to assign a Klimisch reliability score to these studies since they were obtained from a secondary source. However, the Testing Group believes the reported results are

consistent and the information is of sufficient quality and detail to allow it to be used to fulfill the data needs for the *in vitro* genotoxicity endpoint on the residual base oils subcategory.

***In-Vivo* (Chromosomal Aberrations)**

Distillate base oils

Unrefined & Mildly Refined

There are no *in vivo* genotoxicity data available for the unrefined base oils. *In vitro* assays (modified Ames) on a number of unrefined or poorly refined base oils have found a strong correlation between an oil's mutagenicity potential and its 3-7 ring PAC content (Blackburn, et al., 1986; Roy, et al., 1988).

The Testing Group expects the same correlation to exist for *in vivo* genotoxicity and expects unrefined & mildly refined base oils would be active in such studies. Consequently, the Testing Group is not proposing *in vivo* genotoxicity testing of unrefined & mildly refined base oils.

Highly & Severely Refined

A total of seven base stocks were tested in male and female Sprague-Dawley rats using a bone marrow cytogenetics assay (Conaway, et al., 1984). The test materials were administered via gavage at dose levels ranging from 500 to 5000 mg/kg (bw). Dosing occurred for either a single day or for five consecutive days. None of the base oils produced a significant increase in aberrant cells. The Testing Group is unable to assign a Klimisch reliability score to the study since it was obtained from a secondary source and does not contain raw data. However, the Testing Group believes the information is consistent with the results of *in vitro* mutagenicity tests on similar base oils and is of sufficient quality and detail to allow it to be used to fulfill the data needs for the *in vivo* genotoxicity endpoint on the "Highly & Severely refined" base oils.

Residual Base Oils

There is no *in vivo* genotoxicity data available for the residual base oils. However, *in vitro* mutagenicity tests have been conducted on residual base oils and have produced negative results. Dermal carcinogenicity studies on these materials have also been negative. Given these consistent results, and the low bioavailability of these materials, the Testing Group expects *in vivo* mutagenicity tests would also be negative. Consequently, the Testing Group is not proposing *in vivo* genotoxicity testing of a residual base oil.

Summary: No additional testing is planned. Existing *in vitro*, *in vivo* and carcinogenicity studies are adequate to characterize the genotoxicity of distillate and residual base oils.

Reproductive/Developmental Toxicity

Distillate Base Oils

Unrefined & Mildly Refined

No reproductive or developmental toxicity studies have been reported for unrefined & mildly refined distillate base oils. However, a developmental toxicity screening study has been reported for heavy vacuum gas oil, a material with a process history similar to the unrefined distillate base oils (Mobil, 1987). As an unrefined vacuum distillate material, heavy vacuum gas oil contains the broadest spectrum of chemical components and highest concentration of bioavailable and/or biologically active components of all the materials addressed in this Test Plan. Because of their lack of or low level of processing, in comparison to other category members the unrefined lubricating base oils will also have higher concentrations of bioavailable and/or biologically active components.

In the Mobil study, heavy vacuum gas oil was applied daily to the skin of pregnant rats on days 0-19 of gestation. Dose levels administered included: 30, 125, 500 and 1000 mg/kg (bw)/day. All animals were euthanized on day 20. In the dams, the only dose-related finding at gross necropsy was pale colored lungs in four animals in the highest dose group and in one animal in the 500 mg/kg (bw)/day group. Mean thymus weights of the dams in the highest dose group were approximately half those of the control groups. Although absolute liver weights were unaffected by exposure to the gas oil, mean relative liver weights were increased (approximately 15%) in groups exposed to doses greater than 125 mg/kg (bw)/day. Maternal and fetal body weights were reduced at 500 and 1000 mg/kg (bw)/day. Significant increases in resorptions were also seen in these two dose groups. Soft tissue variations and malformations, and skeletal malformations were also increased at 500 and 1000 mg/kg (bw)/day.

Highly & Severely Refined

In the three studies summarized below, highly or severely refined base oils were used as vehicle controls. Consequently, these studies provide limited evidence of the lack of developmental effects of highly & severely refined base oils. Although there were no untreated animals for comparison, the results were considered to be within normal limits.

A highly refined base oil was used as the vehicle control in a one-generation reproduction study (WIL, 1995). The study was conducted according to the OECD Test Guideline 421 "Reproductive/Developmental Toxicity Screening Test". A dose of 1.15 mg/kg (bw) of the base oil was administered daily by gavage to male and female Sprague Dawley rats. Rats were dosed for a minimum of 14 days prior to mating. Dosing was continued after mating until a total dosing period of 30 days had elapsed for males and until day 4 of lactation for females (39 days). There were no clinical findings. Growth rates and food consumption values were normal. There was no effect on fertility and mating indices in either males or females. At necropsy, there were no consistent findings and organ weights and histopathology were considered normal by the study's authors.

McKee, et al. (1987b) reported on a single generation study in which a white mineral oil (a food/drug grade severely refined base oil) was used as a vehicle control. Each of the two separate vehicle control groups contained male and female Sprague-Dawley rats. The dosed animals were given a single daily dose of 5 ml/kg (bw) of the base oil. Dosing was done via gavage, 5 days/week for 13 weeks. After 13 weeks of dosing, the animals were mated. The mated females were maintained without further dosing through gestation and lactation to post-partum day 21. Gross observations of pups and dams were generally unremarkable. In one base oil group, 3 malformed pups were found amongst 2 litters. Two of the malformed pups had syndactyly and renal agenesis, one of the pups also exhibited agnathia. The third pup had a small eye. In the other base oil group, four malformed pups were found amongst four litters. Two of the pups had tail abnormalities, one had a depression in the sternum and the fourth had a short snout. The study authors noted that a similar spectrum of malformations in Sprague-Dawley rats from the same supplier has been reported elsewhere. The authors also commented that this spectrum of malformations occurs spontaneously in the Sprague-Dawley rat.

A white mineral oil (food/drug grade severely refined base oil) was also used as a vehicle control in a developmental toxicity study in Sprague Dawley rats reported by McKee, et al., (1987a). Two separate groups of pregnant rats were administered 5 ml/kg (bw)/day of the base oil via gavage, on days 6 through 19 of gestation. In one of the two base oil dose groups, three malformed fetuses were found among three litters. One fetus had an extra lumbar vertebra, one had a discrete area of ossification in the area of the junction of the frontal and nasal bones, and the third had moderately dilated lateral ventricles of the brain. Three malformed fetuses were also found amongst three litters of the other base oil dose group. Two of these three fetuses had a vertebral arterial canal of a cervical process fully ossified, while the third fetus had angulated ribs. The study authors considered these malformations to be minor and within the normal ranges for the strain of rat.

Residual base oils

There are no reproductive or developmental toxicity data available for the residual base oils.

Summary: A representative sample of a highly to severely refined base oil (other than a food/drug grade white mineral oil) will be tested via the dermal route in a reproductive/developmental screening study (OECD 421). A representative sample of a residual base oil will be tested via the dermal route using a 28-day combined repeated-dose/reproductive/developmental toxicity screening protocol (OECD Test Guideline 422). The limited reproductive and developmental data for highly & severely refined distillate base oils, coupled with positive effects for this endpoint in a study of heavy vacuum gas oil (a material similar to an unrefined distillate base oil) suggests the need for additional reproductive/developmental toxicity data on the highly & severely refined distillate oils. If the test of the highly to severely refined non-food/drug grade base oil is positive, the Testing Group will consider the need to perform a similar test on a food/drug grade white mineral oil. The lack of reproductive and developmental toxicity studies on residual base oils suggests the need for additional reproductive and developmental data on these materials. The Testing Group is not recommending testing of an unrefined to mildly refined lubricating base oil. The Testing Group believes the reproductive and developmental effects of the unrefined & mildly refined base oils are adequately addressed by data on heavy vacuum gas oil.

Evaluation of Existing Physicochemical and Environmental Fate Data

Physicochemical Data

Although some data for products in this category exist, not all of these endpoints are defined and a consensus database for chemicals that represent products in this category does not exist. Therefore, calculated and measured representative data have been identified and a technical discussion provided, where appropriate. The EPIWIN[®] computer model, as discussed in the US EPA document entitled "The Use of Structure-Activity Relationships (SAR) in the High Production Volume Chemicals Challenge Program" has been used to calculate physical-chemical properties of representative constituents of lubricating base oils (U.S. EPA, 2000).

Because of the diversity of compounds encompassing lubricating base oils, it is not feasible to model the physicochemical endpoints for each potential compound. Rather, modeling efforts were directed towards those hydrocarbon components of the base oils that would most likely be dispersed to various environmental media. Since molecular weight and structural conformation determine in large part the solubility and vapor pressure characteristics of the hydrocarbons, modeling focused on representative lower molecular weight hydrocarbons (paraffinic, naphthenic and aromatic). The C15 hydrocarbons were selected since they are the shortest carbon chain length compounds in the base oils, which consist primarily of C15 to C50 compounds (CONCAWE, 1997).

Melting Point

For complex mixtures like petroleum products melting point may be characterized by a range of temperatures reflecting the melting points of the individual components. To better describe the physical phase or flow characteristics of petroleum products, the pour point is routinely used. The pour point is the lowest temperature at which movement of the test specimen is observed under prescribed conditions of the test (ASTM, 2002a). The pour point increases as an oil's viscosity increases. Pour point values for a variety of lubricating base oils have been reported in the literature (Doak, 1983; CONCAWE, 1984; 1993; 1997; Baker, 1984; Singh, et al. 1987; Sequeira, 1992; Montanari, et al., 1998). For example, the pour points measured for eight various unrefined and highly refined base oils ranged from -60 °C to -6 °C (CONCAWE, 1997).

Summary: No additional testing is proposed. The pour point of various lubricating base oils has been adequately measured.

Boiling Point

Because they are mixtures, lubricating base oils do not have a single numerical value for boiling point, but rather a boiling range that reflects the individual components. Constituent hydrocarbons of oils produced from vacuum distillation have boiling points ranging from about 300 to 800°C (CONCAWE, 1997). Boiling ranges for a variety of lubricating base oils have been reported (CONCAWE, 1984; McKee, et al., 1989; Skisak, et al., 1994; Kramer, 1999). For example, distillation ranges have been reported for three lubricating base oil refinery streams; 313 to 432 °C (unrefined, light paraffinic distillate: CAS No. 64741-50-0), 232 to 418 °C (hydrotreated light naphthenic; CAS No. 64742-53-6), and 338 to 604 °C (hydrotreated heavy naphthenic; CAS No. 64742-52-5) (API, 1987b).

Summary: No additional testing is proposed. The boiling range of lubricating base oils has been adequately addressed.

Vapor Pressure

Vapor pressures of lubricating base oils are reported to be negligible (CONCAWE, 1997). In one study, the experimentally measured vapor pressure of a solvent-dewaxed heavy paraffinic distillate base oil was 1.7×10^{-4} Pa (Hazelton UK, 1991). For mixtures such as petroleum products, vapor pressure of the mixture is the sum of the partial pressures of the individual components (Dalton's Law of Partial Pressures). The vapor pressures of base oils cannot be measured experimentally due to analytical limitations for vapor pressure less than 10^{-5} Pa. However, OECD Guideline 104, "Vapor Pressure" (1995), cites seven methods for determining vapor pressure (sensitivities ranging from 10^{-5} to 10^5 Pa), including an estimation method. Therefore, the Testing Group estimated the vapor pressures for base oils with a modified Grain method using the EPIWIN computer software. Since base oils are mixtures of C15 to C50 paraffinic, naphthenic, and aromatic hydrocarbon isomers, representative components of those structures were selected to calculate a range of vapor pressures. The estimated vapor pressure values for these selected components of base oils ranged from 4.5×10^{-1} Pa to 2×10^{-13} Pa. Based on Dalton's Law the expected total vapor pressure for base oils would fall well below minimum levels (10^{-5} Pa) of recommended experimental procedures.

Summary: No additional testing is proposed. The vapor pressures of lubricating base oils are expected to be negligible and have been determined in one study to be 1.7×10^{-4} Pa.

Partition Coefficient (Log K_{ow})

In mixtures such as the base oils, the percent distribution of the hydrocarbon groups (i.e., paraffins, naphthenes, and aromatics) and the carbon chain lengths determines in-part the partitioning characteristics of the mixture. Generally, hydrocarbon chains with fewer carbon atoms tend to have lower partition coefficients than those with higher carbon numbers (CONCAWE, 2001). However, due to their complex composition, unequivocal determination of the log K_{ow} of these hydrocarbon mixtures cannot be made. Rather, partition coefficients of selected C15 chain-length hydrocarbon structures representing paraffinic, naphthenic, and aromatic constituents in base oil lubricants were modeled using the EPIWIN[®], WSKOW V1.40 computer model (U.S. EPA, 2000). Results showed typical log K_{ow} values from 4.9 and higher, which were consistent with values of >4 for lubricating oil basestocks reported by CONCAWE (CONCAWE, 1997).

Summary: No additional modeling is proposed. Partition coefficients (K_{ow}) of 4.9 to 7.7 have been calculated for representative C15 hydrocarbon components of lubricating base oils.

Water Solubility

When released to water, base oils will float and spread at a rate that is viscosity dependent. While water solubility of base oils is typically very low, individual hydrocarbons exhibit a wide range of solubility depending on molecular weight and degree of unsaturation (CONCAWE, 2001). Decreasing molecular weight (i.e., carbon number) and increasing levels of unsaturation increases the water solubility of these materials. As noted for partition coefficient, the water solubility of lubricating base oils cannot be determined due to their complex mixture characteristics. Therefore, the water solubility of individual C15 hydrocarbons representing the different groups making up base oils (i.e., linear and branched paraffins, naphthenes, and aromatics) was modeled using WSKOW V1.40. Based on water solubility modeling of those groups, aqueous solubilities are typically much less than 1 ppm.

Summary: No additional modeling is proposed. Water solubility values of 0.003 to 0.63 mg/L have been calculated for representative C15 hydrocarbon components of lubricating base oils.

Environmental Fate Data

Because the materials included in this category are complex mixtures of differing compositions, it is not possible to measure or calculate a single numerical value for several of the environmental fate properties. Rather, these properties are defined by the range of individual hydrocarbon compounds in the lubricating base oils. The typical battery of tests used to measure the environmental fate of a material is not easily performed on the materials of this category because of their physical and chemical properties. Therefore, components of the lubricating base oils will be modeled where necessary using EPIWIN® (U.S. EPA, 2000).

Photodegradation

Chemicals having potential to photolyze have UV/visible absorption maxima in the range of 290 to 800 nm. Some chemicals have absorption maxima significantly below 290 nm and consequently cannot undergo direct photolysis in sunlight (e.g. chemicals such as alkanes, alkenes, alkynes, saturated alcohols, and saturated acids). Most hydrocarbon constituents of the materials in this category are not expected to photolyze since they do not show absorbance within the 290-800 nm range. However, photodegradation of PAHs can occur and may be a significant degradation pathway for these constituents of lubricating base oils. The degree and rate at which PAHs may photodegrade depend upon whether conditions allow penetration of light with sufficient energy to effect a change. For example, PAC compounds bound to sediments may persist due to a lack of sufficient light penetration.

Atmospheric gas-phase reactions can occur between organic chemicals and reactive molecules such as photochemically produced hydroxyl radicals, ozone and nitrogen oxides. Atmospheric oxidation as a result of radical attack is not direct photochemical degradation, but indirect degradation. In general, lubricating base oils have low vapor pressures and volatilization is not expected to be a significant removal mechanism for the majority of the hydrocarbon components. However, some components (e.g., C15 branched paraffins and naphthenes) appear to have the potential to volatilize. In order to estimate the range of vapor-phase reactivity, calculation of atmospheric oxidation potential (AOP) was applied to specific C15 hydrocarbon components of lubricating base oils. The AOP was determined using the EPIWIN® model, AOPWIN V1.90 (U.S. EPA, 2000).

Summary: No additional modeling is proposed. Atmospheric half-lives of 0.10 to 0.66 days have been calculated for representative C15 hydrocarbon components of lubricating base oils.

Stability in Water

Chemicals that have a potential to hydrolyze include alkyl halides, amides, carbamates, carboxylic acid esters and lactones, epoxides, phosphate esters, and sulfonic acid esters (Harris, 1982). Because lubricating base oils do not contain significant levels of these functional groups, materials in the lubricating base oils category are not subject to hydrolysis.

Summary: Computer modeling will not be conducted for materials in the lubricating base oils category because they do not undergo hydrolysis.

Chemical Transport and Distribution in the Environment (Fugacity Modeling)

Fugacity-based multimedia modeling provides basic information on the relative distribution of chemicals between selected environmental compartments (e.g., air, water, soil, sediment, suspended sediment and biota). The US EPA has agreed that computer-modeling techniques are an appropriate approach to estimating chemical partitioning (fugacity is a calculated, not measured endpoint). A widely used fugacity model is the EQC (Equilibrium Criterion) model (Trent University, 1999). The EQC model is a Level 1 (i.e., steady state, equilibrium, closed system and no degradation) model that utilizes the input of basic chemical properties including molecular weight, vapor pressure, and water solubility to calculate distribution within a standardized regional environment. EPA cites the use of this model in its document "Determining the Adequacy of Existing Data" that was prepared as guidance for the HPV chemicals program (U.S. EPA, 1999).

Based on the physical-chemical characteristics of component hydrocarbons in lubricating base oils, the lower molecular weight components are expected to have the highest vapor pressures and water solubilities, and the lowest partition coefficients. These factors enhance the potential for widespread distribution in the environment. To gain an understanding of the potential transport and distribution of lubricating base oil components, the EQC model was used to characterize the environmental distribution of different C15 compounds representing different structures found in lube oils (e.g., paraffins, naphthenes, and aromatics). The modeling found partitioning to soil or air is the ultimate fate of these C15 compounds. Aromatic compounds partition principally to soil. Linear paraffins partition mostly to soil, while branching appears to allow greater distribution to air. Naphthenes distribute to both soil and air, with increasing proportions in soil for components with the greater number of ring structures. Because Level 1 fugacity modeling does not take into account degradation factors, levels modeled in the atmosphere are likely overstated in light of the tendency for indirect photodegradation to occur. Detailed results of this modeling, including a graphic representation, can be found in section 3.3.1 of the attached Robust Summary.

Summary: No further modeling is proposed. Fugacity-based computer modeling has been done for representative C15 hydrocarbon components of lubricating base oils.

Biodegradation

Twenty-eight biodegradability studies have been reported for a variety of lubricating base oils. Based on the results of ultimate biodegradability tests using modified Sturm and manometric respirometry testing the base oils are expected to be, for the most part, inherently biodegradable. Biodegradation rates found using the modified Sturm procedure ranged from 1.5 to 29%. Results from the manometric respirometry tests on similar materials showed biodegradation rates from 31 to 50%. Biodegradation rates measured in 21-day CEC tests for similar materials ranged from 13 to 79% (BP International Ltd., 1990p-y; 1991a-k; Exxon Biomedical Sciences, Inc., 1995a-d; Shell Research Ltd., 1986; 1987).

The biodegradation rates given above are supported by the conclusions of a CONCAWE review of the biodegradation data for lubricating base oils (CONCAWE, 1997). The CONCAWE review concluded that the extent of biodegradation measured for a particular lubricating oil basestock is dependent not only on the procedure used but also on how the sample is presented in the biodegradation test. In spite of the presentation method, CONCAWE reported that lubricant base oils typically are not readily biodegradable in standard 28-day tests. However, since the oils consist primarily of hydrocarbons that are ultimately assimilated by microorganisms, CONCAWE considered the oils to be inherently biodegradable.

Summary: No additional testing is proposed. Sufficient data exists to characterize the biodegradability of the lubricating base oils.

EVALUATION OF EXISTING ECOTOXICITY DATA AND PROPOSED TESTING

Numerous acute studies covering fish, invertebrates, and algae have been conducted to assess the ecotoxicity of various lubricating base oils. None of these studies have shown evidence of acute toxicity to aquatic organisms. Eight, 7-day exposure studies using rainbow trout failed to demonstrate toxicity

when tested up to the maximum concentration of 1000 mg/L applied as dispersions (BP International Ltd., 1990a-h). Three, 96-hour tests with rainbow trout (BP International Ltd., 1990i-k) also failed to show any toxic effects when tested up to 1000 mg/L applied as dispersions. Similarly, three 96-hour tests with fathead minnows at a maximum test concentration of 100 mg/L water accommodated fractions (WAF) (Exxon Biomedical Sciences Inc. 1995e-g) showed no adverse effects. Two species of aquatic invertebrates (*Daphnia magna* and *Gammarus* sp.) were exposed to WAF solutions up to 10,000 mg/L for 48 and 96-hours, respectively, with no adverse effects being observed (Shell Research Ltd., 1988). Four-day exposures of the freshwater green alga (*Scenedesmus subspicatus*) to 500 mg/L WAF solutions failed to show adverse effects on growth rate and algal cell densities in four studies (BP International Ltd., 1990l-o).

Multiple chronic ecotoxicity studies have shown no adverse effects to daphnid survival or reproduction. In 10 of 11 chronic studies, daphnids were exposed for 21 days to WAF preparations of lubricating base oils with no ill effects on survival or reproduction at the maximum concentration of 1000 mg/L (BP Oil Europe, 1995a-g; Shell Research Ltd., 1994, 1995). One test detected a reduction in reproduction at 1000 mg/L (Shell Research Ltd., 1995). Additional data provided in CONCAWE (1997) support findings of no chronic toxicity to aquatic invertebrates and fish. No observed effect levels ranged from 550 to 5,000 mg/L when tested as either dispersions or WAFs.

The data described above are supported by studies on a homologous series of alkanes reported by Adema (1986). The author concluded that the water solubility of carbon chains $\geq C_{10}$ is too limited to elicit acute toxicity. This also was shown for alkylbenzene compounds having carbon numbers $\geq C_{15}$. Since base oils consist of carbon compounds of C_{15} to C_{50} , component hydrocarbons that are of acute toxicological concern are, for the most part, absent in these materials. Similarly, due to their low solubility, the alkylated two to three ring polyaromatic components in base oils are not expected to cause acute or chronic toxicity. This lack of toxicity is borne out in the results of the reported studies.

Summary: No ecotoxicity testing is proposed. Sufficient data exists to characterize the ecotoxicity of the lubricating base oils.

Data availability for the two subcategories of lubricating oil basestocks is summarized in the following table.

Table 3. Matrix of Available Data and Proposed Testing

| TEST | DISTILLATE BASE OILS | | RESIDUAL BASE OILS |
|-------------------------------------|----------------------------|---------------------------|--------------------------|
| | UNREFINED & MILDLY REFINED | HIGHLY & SEVERELY REFINED | |
| Physical/Chemical Properties | | | |
| Melting Point | Adequate | Adequate | Adequate |
| Boiling Point | Adequate | Adequate | Adequate |
| Vapor Pressure | Adequate | Adequate | Adequate |
| Water Solubility | Adequate | Adequate | Adequate |
| Partition coefficient (log Kow) | Adequate | Adequate | Adequate |
| | | | |
| Ecotoxicity | | | |
| Algae Growth Inhibition | Adequate | Adequate | Adequate |
| Acute Freshwater Invertebrate | Adequate | Adequate | Adequate |
| Acute Freshwater Fish | Adequate | Adequate | Adequate |
| | | | |
| Environmental Fate | | | |
| Biodegradation | Adequate | Adequate | Adequate |
| Stability in Water | N/A | N/A | N/A |
| Photodegradation (estimate) | Adequate | Adequate | Adequate |
| Transport and Distribution | Adequate | Adequate | Adequate |
| | | | |
| Mammalian Toxicity | | | |
| Acute | Adequate | Adequate | Adequate |
| Repeat-dose | Adequate | Adequate | Test |
| Reprod/Develop | Read Across ¹ | Test | Test |
| Genotoxicity, <i>in vitro</i> | Adequate | Adequate | Adequate |
| Genotoxicity, <i>in-vivo</i> | Read Across ² | Adequate | Read Across ³ |

¹ Read Across from existing study on heavy vacuum gas oil.

² Read Across from *in vitro* and carcinogenicity data on unrefined & mildly refined base oils.

³ Read Across from *in vitro* and carcinogenicity data on residual base oils.

Adequate: Indicates adequate existing data.

N/A: Indicates that evaluation of endpoint is Not Applicable due to physical-chemical properties

References

Adema, D.M.M. and G.H. van den Bos Bakker. 1986. Aquatic toxicity of compounds that may be carried by ships (Marpol 1973, Annex II) – a progress report for 1986 from TNO to the Dutch Ministry of Housing, Physical Planning and Environment. Report no. 86/326a. Delft: TNO [as cited in CONCAWE 1997].

API (American Petroleum Institute). 1986a. 28 day dermal toxicity study in the rabbit API 83-12 Hydrotreated light naphthenic distillate CAS 64742-53-6 Study conducted by Tegriss Laboratories Inc. API Med. Res. Publ. 33-30499 Washington DC.:American Petroleum Institute.

API (American Petroleum Institute). 1986b. 28 day dermal toxicity study in the rabbit API 84-01 Light paraffinic distillate CAS 64741-50-0 Study conducted by Tegriss Laboratories Inc. API Med. Res. Publ. 33-31642 Washington DC.:American Petroleum Institute.

API (American Petroleum Institute). 1986c. Acute oral toxicity study in rats Acute dermal toxicity study in rabbits Primary dermal irritation study in rabbits Primary eye irritation study in rabbits Dermal sensitization study in Guinea pigs API 83-12 Hydrotreated light naphthenic distillate CAS 64742-53-6 Study conducted by Hazleton Laboratories Inc. API Med. Res. Publ.: 33-30592 Washington DC: American Petroleum Institute.

API (American Petroleum Institute). 1986d. Acute oral toxicity study in rats Acute dermal toxicity study in rabbits Primary dermal irritation study in rabbits Primary eye irritation study in rabbits Dermal sensitization study in Guinea pigs API 84-01 Light paraffinic distillate CAS 64741-50-0 Study conducted by Hazleton Laboratories Inc. API Med. Res. Publ.: 33-30595 Washington DC: American Petroleum Institute.

API (American Petroleum Institute). 1987a. Acute inhalation toxicity evaluation of a petroleum derived hydrocarbon in rats. API 83-12 Hydrotreated light naphthenic distillate CAS 64742-53-6 Study conducted by Hazleton Laboratories America Inc. API HESD Publ. 34-32775 Washington DC: American Petroleum Institute.

API (American Petroleum Institute). 1987b. Comprehensive Analytical Analysis of API Generic Refinery Streams. API, Washington, DC. : American Petroleum Institute.

API (American Petroleum Institute) 1992. Mineral Oil Review. Health & Environmental Sciences, API Departmental Report Number DR 21; January; Order No. 848-00021 Washington DC: American Petroleum Institute.

ASTM (American Society for Testing and Materials). 2002a. D97-02 standard test method for pour point of petroleum oils. West Conshohocken, PA.

ASTM (American Society for Testing and Materials) 2002b. E1687-98 standard test method for determining carcinogenic potential of virgin base oils in metalworking fluids. West Conshohocken, PA.

Baker, A.E. 1984. Lubricant Properties and Test Methods, Handbook of Lubrication, Theory, and Practice of Tribology, Vol I, ed E.R. Booser, CRC Press.

BIBRA 1992. A 90-day feeding study in the rat with six different mineral oils (N15(H), N70(H), N70(A), P15(H), N10(A) and P100(H), three different mineral waxes (a low melting point wax, a high melting point wax and a high sulphur wax) and coconut oil. BIBRA project No. 3.1010

Bingham, E. Trosset, R. P., Warshawsky, D. 1980. Carcinogenic potential of petroleum hydrocarbons, a critical review of the literature. J. Environmental Pathology and Toxicology, Vol 3, pp 483-563.

Bird, M. G., Lewis, S. C., Freeman, J. J., Smith, J. H., Hogan, G. K and Scala, R. A. 1990. Subchronic feeding study of white mineral oils in rats and dogs. Toxicologist Vol. 10, No 1, page 143.

Blackburn, G.R., Deitch, R.A., Schreiner, C.A., Mehlman, M. A. and Mackerer, C.R. 1984. Estimation of the dermal carcinogenic activity of petroleum fractions using a modified Ames assay. Cell Biol. and Toxicol. Vol 1, No 1, pp 67-80.

Blackburn, G. R., Deitch, R. A., Schreiner, C. A. And Mackerer, C. R. 1986. Predicting tumorigenicity of petroleum distillation fractions using a modified Salmonella Mutagenicity assay. Cell Biol. Toxicol. Vol. 2. pp 63-84.

Blackburn, G.R., Roy, T.A., Bleicher, W.T., Reddy, V.M., Mackerer, C.R. 1996. Comparison of Biological and Chemical Predictors of Dermal Carcinogenicity of Petroleum Oils, Polycyclic Aromatic Hydrocarbons 11:201–208.

BP International Limited. 1990a. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/1; Report No. AT301-021.

BP International Limited. 1990b. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/2; Report No. AT301/022.

BP International Limited. 1990c. The Acute Toxicity of to Rainbow Trout (*Salmo gairdneri*). Project No. 301/3; Report No. AT301/023.

BP International Limited. 1990d. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/4; Report No. AT301/024.

BP International Limited. 1990e. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/5; Report No. AT301/025.

BP International Limited. 1990f. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/6; Report No. AT301/026.

BP International Limited. 1990g. The Acute Toxicity of to Rainbow Trout (*Salmo gairdneri*). Project No. 301/7; Report No. AT301/027.

BP International Limited. 1990h. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/8; Report No. AT301/028.

BP International Limited. 1990i. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/55; Report No. AT301/042.

BP International Limited. 1990j. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/56; Report No. AT301/043R.

BP International Limited. 1990k. The Acute Toxicity to Rainbow Trout (*Salmo gairdneri*). Project No. 301/65; Report No. AT301/044.

BP International Limited. 1990l. Assessment of the Algistatic Effect of **** to *Scenedesmus subspicatus*. Project No. 301/70.

BP International Limited. 1990m. Assessment of the Algistatic Effect of **** to *Scenedesmus subspicatus*. Project No. 301/72.

BP International Limited. 1990n. Assessment of the Algistatic Effect of **** to *Scenedesmus subspicatus*. Project No. 301/74.

BP International Limited. 1990o. Assessment of the Algistatic Effect of **** to *Scenedesmus subspicatus*. Project No. 301/76.

BP International Limited. 1990p. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/10; Report No. AT301/030.

BP International Limited. 1990q. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/11; Report No. AT301/031.

BP International Limited. 1990r. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/12; Report No. AT301/034.

BP International Limited. 1990s. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/13; Report No. AT301/032

BP International Limited. 1990t. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/15; Report No. AT301/035.

BP International Limited. 1990u. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/16; Report No. AT301/036.

BP International Limited. 1990v. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/60; Report No. AT301/038.

BP International Limited. 1990w. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/64; Report No. AT301/064.

BP International Limited. 1990x. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/9; Report No. AT301/029.

BP International Limited. 1990y. Assessment of Ready Biodegradability (Modified Sturm Test). Project No. 301/59; Report No. AT301/037.

BP International Limited. 1991a. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3823/B, Performing Laboratory Study No. T119/A.

BP International Limited. 1991b. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3820/B, Performing Laboratory Study No. T116/A.

BP International Limited. 1991c. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3824/B, Performing Laboratory Study No. T120/A.

BP International Limited. 1991d. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3825/B, Performing Laboratory Study No. T121/A.

BP International Limited. 1991e. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3970/B, Performing Laboratory Study No. T651/A.

BP International Limited. 1991f. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3971/B, Performing Laboratory Study No. T652/A.

BP International Limited. 1991g. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3975/B, Performing Laboratory Study No. T930/A.

BP International Limited. 1991h. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3819/B, Performing Laboratory Study No. T115/A.

BP International Limited. 1991i. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3821/B, Performing Laboratory Study No. T117/A.

BP International Limited. 1991j. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3822/B, Performing Laboratory Study No. T118/A.

BP International Limited. 1991k. Mineral Hydrocarbon Oil: Biodegradability by CEC Method L-33-T-82. Report No. BL3826/B, Performing Laboratory Study No. T122/A.

BP Oil Europe. 1995a. Daphnia magna Reproduction Test. SPL Project No. 692/036.

BP Oil Europe. 1995b. Daphnia magna Reproduction Test. SPL Project No. 692/037.

BP Oil Europe. 1995c. Daphnia magna Reproduction Test. SPL Project No. 692/038.

BP Oil Europe. 1995d. Daphnia magna Reproduction Test. SPL Project No. 692/039.

BP Oil Europe. 1995e. Daphnia magna Reproduction Test. SPL Project No. 692/040.

BP Oil Europe. 1995f. Daphnia magna Reproduction Test. SPL Project No. 692/041.

BP Oil Europe. 1995g. Daphnia magna Reproduction Test. SPL Project No. 692/042.

Chasey, K.L. and McKee, R.H. 1993. Evaluation of the Dermal Carcinogenicity of Lubricant Base Oils by the Mouse Skin Painting Bioassay and Other Proposed Methods. J. of Appl. Tox. 13(1): 57-65.

Conaway, C. C., Schreiner, C. A. and Cragg, S. T. 1984. Mutagenicity evaluation of petroleum hydrocarbons In: Advances in modern experimental toxicology Volume VI: Applied toxicology of hydrocarbons, pp 89-107. Eds MacFarland et al., Princeton Scientific Publishers.

CONCAWE. 1984. Assessment and comparison of the composition of food-grade white oils and waxes manufactured from petroleum by catalytic hydrogenation versus conventional treatment. Report No. 84/60, Brussels.

CONCAWE. 1993. White Oil and Waxes Summary of 90-Day Studies. Report No. 93/56, Brussels.

CONCAWE. 1994. The use of the dimethyl sulphoxide (DMSO) extract by the IP 346 method as an indicator of the carcinogenicity of lubricant base oils and distillate aromatic extracts. Report No. 94/51, Brussels.

CONCAWE. 1997. Lubricating Oil Basestocks. Product Dossier No. 97/108, Brussels.

CONCAWE. 2001. Environmental Classification of Petroleum Substances – Summary Data and Rationale. Report No. 01/54, Brussels.

Dalbey, W., Osimitz, T., Kommineni, C., Roy, T., Feuston, M., and Yang, J. 1991. Four-week inhalation exposures of rats to aerosols of three lubricant base oils J. Appl. Toxicol. Vol 11 (4), pp 297-302.

Doak, S.M.A., Brown, V.K.H., Hunt, P.F., Smith, J.D., and Roe, R.J.C. 1983. The carcinogenic potential of twelve refined mineral oils following long-term topical application. Br. J. Cancer 48, pp 429-436.

Eisler, R. 1987. Polycyclic aromatic hydrocarbon hazards to fish, wildlife, and invertebrates: a synoptic review. U.S. Fish and Wildlife Service Biological Report 85(1.11). 81 pp.

EU (European Union) 1994. Commission Directive 94/69/EC of 19 December 1994 adapting to technical progress for the 21st time Council Directive 67/548/EEC on the approximation of the laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances. Official Journal of the European Communities No L381, 31.12.

EMBSI (ExxonMobil Biomedical Sciences, Inc.) 2000. 00MRL 18.

EMBSI (ExxonMobil Biomedical Sciences, Inc.) 2001a. 01.MRL.66.

EMBSI (ExxonMobil Biomedical Sciences, Inc.) 2001b. Combined chronic toxicity/carcinogenicity study of white oil in Fischer 344 rats. Test substance 70cSt White oil. Study performed for CONCAWE Project No. 105970 Exxon Biomedical Sciences Inc. New Jersey July 11.

Exxon 1984. Internal Report REHD (MR.32DO.84).

Exxon Biomedical Sciences, Inc. 1995a. Ready Biodegradability, Manometric Respirometry. Study #107194A.

Exxon Biomedical Sciences, Inc. 1995b. Ready Biodegradability, Manometric Respirometry. Study #123694A.

Exxon Biomedical Sciences, Inc. 1995c. Ready Biodegradability, Manometric Respirometry. Study #107094A.

Exxon Biomedical Sciences, Inc. 1995d. Ready Biodegradability, Manometric Respirometry. Study #198194A.

Exxon Biomedical Sciences, Inc. 1995e. Fathead Minnow Acute Fish Toxicity Test. Study #101740.

Exxon Biomedical Sciences, Inc. 1995f. Fathead Minnow Acute Fish Toxicity Test. Study #198140.

Exxon Biomedical Sciences, Inc. 1995g. Fathead Minnow Acute Fish Toxicity Test. Study #198240.

Feuston, M.H., Low, L.K., Hamilton, and C.E., Mackerer, C.R. 1994. Correlation of systemic and developmental toxicities with chemical component classes of refinery streams. Fund. Appl. Toxicol. 22:622-630.

Firriolo, J. M., Morris, C. F., Trimmer, G. W., Twitty, L. D., Smith, J. H. and Freeman, J. J. 1995. Comparative 90-day feeding study with low-viscosity white mineral oil in Fischer-344 and Sprague-Dawley-derived CRL:CD rats. Toxicologic Pathology Vol 23, No. 1, pages 26-33.

Halder, C.A., Warne, T.M., Little, R.Q., and Garvin, P.J. 1984. Carcinogenicity of Petroleum Lubricating Oil Distillates: Effects of Solvent Refining, Hydroprocessing and Blending. Am. J. of Indust. Med. 5:265-274.

Harris, J.C. 1982. Rate of Hydrolysis. In Handbook of Chemical Property Estimation Methods. p. 7-6. W. J. Lyman, W.F. Reehl and D.H. Rosenblatt, eds. McGraw-Hill Book Company, New York, NY, USA.

Hazleton UK (for Shell Research Ltd.) 1991. Determination of Vapour Pressure. Report No. 6736-579/70.

IARC 1984. IARC Monographs on the evaluation of the carcinogenic risk of chemicals to humans, Volume 33: Polynuclear aromatic hydrocarbons, part 2, carbon blacks, mineral oils (lubricant base oils and derived products) and some nitroarenes. International Agency for Research on Cancer, Lyon.

IP (Institute of Petroleum) 1985. Methods for Analysis and Testing, IP 386/80, polycyclic aromatics in petroleum fractions by dimethyl sulphoxide Vol 2:346.1-346.6.

JECFA (WHO Joint Expert Committee on Food Additives) 1996. Toxicological evaluation of certain food additives and contaminants. Prepared by the 44th meeting of the Joint FAO/WHO Expert Committee on Food Additives (JECFA). WHO Food Additives Series 35. Geneva.

JECFA (WHO Joint Expert Committee on Food Additives) 2002. Toxicological evaluation of certain food additives and contaminants. Prepared by the 59th meeting of the Joint FAO/WHO Expert Committee on Food Additives (JECFA). WHO Food Additives Series. Geneva.

Kane, M.L., Ladov, E.N., Holdsworth, C.E. and Weaver, N.K. 1984. Toxicological characteristics of refinery streams used to manufacture lubricating oils. Am. J. of Indust. Med. 5:183-200.

King, D. J. 1991. 1156, 1157 and 1158: 2-year skin painting study. BP Group Occupational Health Centre, Toxicology report 25-90-0275.

Kramer, D.C., Ziemer, J.N., Cheng, M.T., Fry, C.E., Reynolds, R.N., Lok, B.K., Sztenderowicz, M.L., and Krug, R.R. 1999. Influence of Group II & III Base Oil Composition on VI and Oxidation Stability Presentation at 66th NLGI Annual Meeting, Tucson AZ Oct 24-27.

McKee, R. H., Pasternak, S. J. and Traul, K. A. 1987a. Developmental toxicity of EDS recycle solvent and fuel oil. Toxicology Vol 46, pp 205-215.

McKee, R. H., Plutnick, R. T. and Traul, K. A. 1987b. Assessment of the potential reproductive and subchronic toxicity of EDS coal liquids in Sprague-Dawley rats. Toxicology Vol 46, pp 267-280.

McKee, R.H., Daughtrey, W.C., Freeman, J.J., Federici, T.M., Phillips, R.D., Plutnick, R.T. 1989. The dermal carcinogenic potential of unrefined and hydrotreated lubricating oils. J. Appl. Toxicology, Vol 9(4), 265-170.

Mobil 1997. Petroleum Manufacturing Orientation Course. Mobil Research and Engineering, Paulsboro, NJ.

Mobil 1987. Developmental toxicity screen in rats exposed dermally to heavy vacuum gas oil (HVGO) Study No. 61801 Final report. Mobil Environmental and Health Science Laboratory: Princeton.

Montanari, L., Montani, E., Corno, C., and Fattori, S. 1998. NMR molecular characterization of lubricating base oils: correlation with their performance. Appl. Magn. Reson. 14, pp 345-356.

Petrolabs 1998. H-Mobil-67763-Vacuum Resid. : Pennsylvania

Petrolabs 2000. H-Mobil-68351-Bright stock: Pennsylvania

Roy, T.A., Johnson, S.W., Blackburn, G.R., and Mackerer, C.R. 1988. Correlation of mutagenic and dermal carcinogenic activities of mineral oils with polycyclic aromatic compound content. Fund. Appl. Toxicol. Vol 10, pp 466-476.

Roy, T.A., Blackburn, G.R., and Mackerer, C.R. 1996. Evaluation of physicochemical factors affecting dermal penetration and carcinogenic potency of mineral oils containing polycyclic aromatic compounds. Polycyclic Aromatic Compounds Vol 10 pp 333-342.

SCF (EU Scientific Committee for Food) 1995. Opinion on mineral and synthetic hydrocarbons (expressed on 22 September 1995) CS/ADD/MsAd/132-Final, Brussels, European Commission.

Sequeira, A. 1992. An overview of lube base oil processing. Symposium on Processing, Characterization and Application of Lubricant Base Oils, Amer. Chem. Soc. Meeting; Washington, D.C., Aug 23-28.

Shell Research Ltd. 1986. Base Oils: An Assessment of Ready Biodegradability. Report No. SBGR.86.137.

Shell Research Ltd. 1987. Base Oil: An Assessment of Ready Biodegradability. Report No. SBGR.87.259.

Shell Research Ltd. 1988. Oils: Acute toxicity of four oils to *Daphnia magna* and *Gammarus pulex*. Report SBGR.88.075.

Shell Research Limited. 1994. Chronic toxicity of water-accommodated fractions to *Daphnia magna*. Experiment #5922.

Shell Research Limited. 1995. Chronic toxicity of water accommodated fractions to *Daphnia magna*. Experiment #6215.

Shoda, T, Toyoda, K, Uneyama, C., Takada, K. and Takahashi, M. (1997) Lack of carcinogenicity of medium-viscosity liquid paraffin given in the diet to F344 rats. Food and Chemical Toxicology Vol. 35, pages 1181-1190.

Singer, E.J., Mackerer, C.R., Mekitarian, A.E., Diperna, C.J. 1986. Toxicology Testing of Petroleum Products: The Basis for Managing and Communicating Hazards, SAE Technical Paper Series, no. 861595.

Singh, H., and Gulati, I.B. 1987. Group chemical changes and physical property correlations in refining of lube base stocks. Revue de L'Institut Francais Du Petrole, Vol 42, No. 4 pp 493-504.

Skisak, C.M., Furedi-Machacek, E.M., Schmitt, S.S., Swanson, M.S., and Vernot, E.H. 1994. Chronic and initiation/promotion skin bioassays of petroleum refinery streams. Env. Hlth. Perspect. 102 No. 11 pp 82-87.

Trent University. 1999. Level 1 Fugacity-Based Environmental Equilibrium Partitioning Model, V2.11. Environmental Modelling Centre, Trent University, Canada.

U.S. EPA (Environmental Protection Agency). 1999. Guidance for Assessing Adequacy of Existing Data, <http://www.epa.gov/chemrtk/guidocs.htm>.

U.S. EPA (Environmental Protection Agency). 2000. EPI (Estimation Program Interface) Suite, V3.10. <http://www.epa.gov/opptintr/exposure/docs/episuite.htm>.

U.S. FDA (Food and Drug Administration). 2002. Food Additives Permitted for Direct Addition to Food for Human Consumption Code of Federal Regulation Title 21, Vol 3 Part 172, Subpart I, Section 172.878.

USP (U.S. Pharmacopeia), 2002. United States Pharmacopeia (USP) 25 – National Formulary (NF) 20. U.S. Pharmacopeia, Rockville.

WHO (World Health Organization) 1982. Selected Petroleum Products. Environ. Health Criteria Document No. 20. World Health Organization, Geneva.

WIL Research Laboratories Inc. 1995. An oral reproduction/developmental toxicity screening study of **** in finished oil in rats. Laboratory Study No. WIL-187007: Ohio.

APPENDIX A.

CAS Numbers and Definitions of Category Members

The CAS numbers and definitions of refinery streams, including lubricating oil basestocks, were developed in response to Section 8(b) of the Toxic Substances Control Act. This section of TSCA required identification and registration with the Environmental Protection Agency before July 1979 of each "chemical substance" being manufactured, processed, imported or distributed in commerce. Due to analytical limitations and known variability in refinery stream composition, identification of every specific individual molecular compound in every refinery process stream under all processing conditions was impossible. Recognizing these problems, the American Petroleum Institute (API) recommended to the EPA a list of generic names for refinery streams consistent with industry operations and covering all known processes used by refiners. The list, including generic names, CAS numbers and definition of each stream, was published by the EPA as "Addendum I, Generic Terms Covering Petroleum Refinery Process Streams."

Because of the variability inherent in the processing of petroleum materials, the definitions API developed for the CAS numbers are qualitative in nature, written in broad, general terms. The definitions often contain ranges of values, with little if any quantitative analytical information or concern for possible compositional overlaps. Many of the definitions also include information on the material's process history. In fact, process history and not chemical composition was one of the primary criteria used by API to differentiate streams and assign CAS numbers. In practice, process history was defined as the final process step a refinery stream had undergone. Information on intermediate processing steps was generally not included in the CAS definition. The result is that the CAS definitions for the lubricating oil basestocks often do not provide an accurate assessment of the refining history for a specific stream. For example, the CAS definition of a light paraffinic stream that has been solvent-refined, then clay treated and finally dewaxed will include only information on the final processing step, the dewaxing:

A complex combination of hydrocarbons obtained by removal of normal paraffins from a petroleum fraction by solvent crystallization. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100 °F (19 cSt at 40 °C).

In the list shown below, using the oils CAS definitions, the Testing Group has identified unrefined and residual base oils. Because of the lack of process history detail in the CAS definitions, the Testing Group is unable to assign a degree of refining to many of the remaining distillate base oils.

DISTILLATE BASE OILS

Unrefined & Mildly Refined

CAS Number

64741-50-0

Distillates (petroleum), light paraffinic

A complex combination of hydrocarbons produced by vacuum distillation of the residuum from atmospheric distillation of crude oil. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains a relatively large proportion of saturated aliphatic hydrocarbons normally present in this distillation range of crude oil.

64741-51-1

Distillates (petroleum), heavy paraffinic

A complex combination of hydrocarbons produced by vacuum distillation of the residuum from atmospheric distillation of crude oil. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of at least 100 SUS at 100°F (19cSt at 40°C). It contains a relatively large proportion of saturated aliphatic hydrocarbons.

64741-52-2

Distillates (petroleum), light naphthenic

A complex combination of hydrocarbons produced by vacuum distillation of the residuum from atmospheric distillation of crude oil. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64741-53-3

Distillates (petroleum), heavy naphthenic

A complex combination of hydrocarbons produced by vacuum distillation of the residuum from atmospheric distillation of crude oil. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of at least 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

Highly & Severely Refined

64741-76-0

Distillates (petroleum), heavy hydrocracked

A complex combination of hydrocarbons from the distillation of the products from a hydrocracking process. It consists predominantly of saturated hydrocarbons having carbon numbers in the range of C₁₅ through C₃₉ and boiling in the range of approximately 260°C to 600°C (500°F to 1112°F).

64741-88-4

Distillates (petroleum), solvent-refined heavy paraffinic

A complex combination of hydrocarbons obtained as the raffinate from a solvent extraction process. It consists predominantly of saturated hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of at least 100 SUS at 100°F (19cSt at 40°C).

64741-89-5

Distillates (petroleum), solvent-refined light paraffinic

A complex combination of hydrocarbons obtained as the raffinate from a solvent extraction process. It consists predominantly of saturated hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C).

64741-96-4

Distillates (petroleum), solvent-refined heavy naphthenic

A complex combination of hydrocarbons obtained as the raffinate from a solvent extraction process. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of at least 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64741-97-5

Distillates (petroleum), solvent-refined light naphthenic

A complex combination of hydrocarbons obtained as the raffinate from a solvent extraction process. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-18-3

Distillates (petroleum), acid-treated heavy naphthenic

A complex combination of hydrocarbons obtained as a raffinate from a sulfuric acid treating process. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of at least 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-19-4

Distillates (petroleum), acid-treated light naphthenic

A complex combination of hydrocarbons obtained as a raffinate from a sulfuric acid treating process. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-34-3

Chemically Neutralized Heavy Naphthenic Distillate (petroleum)

A complex combination of hydrocarbons produced by a treating process to remove acidic materials. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of at least 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-35-4

Chemically Neutralized Light Naphthenic Distillate (petroleum)

A complex combination of hydrocarbons produced by a treating process to remove acidic materials. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-37-6

Distillates (petroleum), clay-treated light paraffinic

A complex combination of hydrocarbons resulting from treatment of a petroleum fraction with natural or modified clay in either a contacting or percolation process to remove the trace amounts of polar compounds and impurities present. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains a relatively large proportion of saturated hydrocarbons.

64742-44-5

Distillates (petroleum), hydrotreated heavy naphthenic

A complex combination of hydrocarbons obtained by treating a petroleum fraction with hydrogen in the presence of a catalyst. It consists of hydrocarbons having finished oil of at least 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-52-5

Distillates, (petroleum), hydrotreated heavy naphthenic

A complex combination of hydrocarbons obtained by treating a petroleum fraction with hydrogen in the presence of a catalyst. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil of at least 100 SUS at 100 degree F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-53-6

Distillates (petroleum), hydrotreated light naphthenic

A complex combination of hydrocarbons obtained by treating a petroleum fraction with hydrogen in the presence of a catalyst. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-54-7

Distillates (petroleum), hydrotreated heavy paraffinic

A complex combination of hydrocarbons obtained by treating a petroleum fraction with hydrogen in the presence of a catalyst. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil of at least 100 SUS at 100°F (19cSt at 40°C). It contains a relatively large proportion of saturated hydrocarbons.

64742-55-8

Distillates (petroleum), hydrotreated light paraffinic

A complex combination of hydrocarbons obtained by treating a petroleum fraction with hydrogen in the presence of a catalyst. It consists of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C). It contains a relatively large proportion of saturated hydrocarbons.

64742-56-9

Distillates (petroleum), solvent-dewaxed light paraffinic

A complex combination of hydrocarbons obtained by removal of normal paraffins from a petroleum fraction by solvent crystallization. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C).

64742-63-8

Distillates (petroleum), solvent-dewaxed heavy naphthenic

A complex combination of hydrocarbon obtained by removal of normal paraffins from a petroleum fraction by solvent crystallization. It consists of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil of not less than 100 SUS at 100°F (19cSt at 40°C). It contains relatively few normal paraffins.

64742-65-0

Distillates (petroleum), solvent-dewaxed heavy paraffinic

A complex combination of hydrocarbons obtained by removal of normal paraffins from a petroleum fraction by solvent crystallization. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity not less than 100 SUS at 100°F (19cSt at 40°C).

64742-70-7

Paraffin oils (petroleum), catalytic dewaxed heavy

A complex combination of hydrocarbons obtained from a catalytic dewaxing process. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of at least 100 SUS at 100°F (19cSt at 40°C).

64742-71-8

Paraffin oils (petroleum), catalytic dewaxed light

A complex combination of hydrocarbons obtained from a catalytic dewaxing process. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil with a viscosity of less than 100 SUS at 100°F (19cSt at 40°C).

72623-85-9

Lubricating oils (petroleum), C₂₀₋₅₀, hydrotreated neutral oil-based, high-viscosity

A complex combination of hydrocarbons obtained by treating light vacuum gas oil, heavy vacuum gas oil, and solvent deasphalted residual oil with hydrogen in the presence of a catalyst in a two stage process with dewaxing being carried out between the two states. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil having a viscosity of approximately 112cSt at 40°C. It contains a relatively large proportion of saturated hydrocarbons.

72623-86-0

Lubricating oils (petroleum), C₁₅₋₃₀, hydrotreated neutral oil-based

A complex combination of hydrocarbons obtained by treating light vacuum gas oil and heavy vacuum gas oil with hydrogen in the presence of a catalyst in a two stage process and dewaxing being carried out between the two stages. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₃₀ and produces a finished oil having a viscosity of approximately 15cSt at 40°C. It contains a relatively large proportion of saturated hydrocarbons.

8042-47-5

White mineral oil (petroleum)

A highly refined petroleum mineral oil consisting of a complex combination of hydrocarbons obtained from the intensive treatment of a petroleum fraction with sulphuric acid and oleum, or by hydrogenation, or by a combination of hydrogenation and acid treatment. Additional washing and treating steps may be included in the processing operation. It consists of saturated hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₅₀.

64742-58-1

Lubricating oils, petroleum, hydrotreated spent

A complex combination of hydrocarbons obtained by treating a spent lube oil with hydrogen in the presence of a catalyst. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₁₅ through C₅₀.

64742-67-2

Foots oil, petroleum

A complex combination of hydrocarbons obtained as the oil fraction from a solvent deoiling or a wax sweating process. It consists predominantly of branched chain hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀.

Note from Testing Group ** Foots oil, obtained from the deoiling of wax made from vacuum distillate, is essentially an unrefined base oil, that undergoes similar process as any of the vacuum tower fractions – each step removing impurities and improving product performance.

72623-84-8

Lubricating oils, (petroleum) C15-30, hydrotreated neutral oil based solvent

Residual Base Oils

64741-95-3

Residual oils (petroleum), solvent deasphalted

A complex combination of hydrocarbons obtained as the solvent soluble fraction from C₃ through C₄ solvent deasphalting of a residuum. It consists of hydrocarbons having carbon numbers predominantly higher than C₂₅ and boiling above approximately 400°C (725°F).

72623-83-7

Lubricating oils (petroleum) C₂₅, hydrotreated bright stock-based

A complex combination of hydrocarbons obtained by treating solvent deasphalted residual oil with hydrogen in the presence of a catalyst in two stages with dewaxing carried out between stages. It consists predominantly of hydrocarbons having carbon numbers predominantly greater than C₂₅ and produces a finished oil with a viscosity of approximately 440cSt at 40°C. It contains a relatively large proportion of saturated hydrocarbons.

72623-87-1

Lubricating oils (petroleum), C₂₀₋₅₀, hydrotreated neutral oil-based

A complex combination of hydrocarbons obtained by treating light vacuum gas oil, heavy vacuum gas oil and solvent deasphalted residual oil with hydrogen in the presence of a catalyst in a two stage process with dewaxing being carried out between the two stages. It consists predominantly of hydrocarbons having carbon numbers predominantly in the range of C₂₀ through C₅₀ and produces a finished oil with a viscosity of approximately 32cSt at 40°C. It contains a relatively large proportion of saturated hydrocarbons.

64742-01-4

Residual oils (petroleum), solvent-refined

A complex combination of hydrocarbons obtained as the solvent insoluble fraction from solvent refining of a residuum using a polar organic solvent such as phenol or furfural. It consists of hydrocarbons having carbon numbers predominantly higher than C₂₅ and boiling above approximately 400°C (725°F).

64742-57-0

Residual oils (petroleum), hydrotreated

A complex combination of hydrocarbons obtained by removal of long, branched chain hydrocarbons from a residual oil by solvent crystallization. It consists of hydrocarbons having carbon numbers predominantly greater than C₂₅ and boiling above approximately 400°C (752°F).

64742-62-7

Residual oils, petroleum, solvent-dewaxed

A complex combination of hydrocarbons obtained by removal of long, branched chain hydrocarbons from a residual oil by solvent crystallization. It consists of hydrocarbons having carbon numbers predominantly greater than C₂₅ and boiling above approximately 400 degree C (752°F).

Appendix B.

Links to Additional Resources

Refining Processes: General Descriptions

http://www.chevron.com/about/learning_center/refinery
<http://www.lubrizol.com/lubetheory/default.htm>
<http://www.orionrefining.com/flow.htm>
http://www.osha-slc.gov/dts/osta/otm/otm_toc.html
http://www.shellglobalsolutions.com/base_oils/library/library.htm
<http://www.shell-lubricants.com/learningcenter/aboutoil.html>
http://www.shellus.com/welcome/history/hist_oil_main.html
<http://www.epa.gov/compliance/resources/publications/assistance/sectors/notebooks/petrefsnpt1.pdf>
http://www.mts.net/~dbrad1/base_oil.htm

Petroleum Related Glossaries

http://www.caltex.com.au/products_glo.asp
<http://www.citgo.com/CommunityInvolvement/Classroom/Glossary.jsp>
<http://www.epplp.com/gloss.html>
http://www.prod.exxon.com/exxon_productdata/lube_encyclopedia/
http://www.hellenic-petroleum.gr/english/glossary/gl_main.htm
http://www.prod.exxon.com/exxon_productdata/lube_encyclopedia/
<http://www.oilanalysis.com/dictionary>
<http://www.orionrefining.com/glossary.htm>
<http://www.gedolbear.com/glossary.htm>
http://www.shellglobalsolutions.com/base_oils/glossary/a_g.htm
http://www.ursa-texaco.com/English/glossary_a.html
http://www.eia.doe.gov/pub/oil_gas/petroleum/data_publications/petroleum_marketing_annual/current/pdf/glossary.pdf

Appendix C.
Robust Summary
(Separate document)